

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No. : 10/595,853
Applicant(s) : NOWAK, Rüdiger et al.
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Title : ADHESIVE AND SEALANT SYSTEMS

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Commissioner for Patents
P.O. Box 1450
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DECLARATION UNDER 37 C.F.R. 1.132

The undersigned, Rüdiger Nowak, a co-inventor in the above-identified application, hereby declares and states as follows:

1. He is completely familiar with the contents of the above-identified application and is advised that the Examiner in the United States Patent and Trademark Office has rejected the claims in this application in view of his prior application which is published application US2001/0047047 A1, ('047) and in which he is a co-inventor with Helmut Steinbach and Adolf Stender. He has again read both the above-identified application 10/595,853, as well as his above-mentioned prior '047 application.

2. In the table on page 2 of the published application '047 under paragraph [0018], there is shown the tests of the silica products known as AEROSIL R202, AEROSIL R805 and AEROSIL R812. Each of these are commercial silica products provided by Evonik Degussa GmbH, the assignee of this application. Each of these silicas exhibit a tamped density of 50 g/l. as shown in the table.

3. The density values reported in the table in paragraph [0018] of the '047 application were obtained by the method known as the "Carter-Filler" method which is the standard densification method used in the silica industry and described in US Patent 4,770,214 as "The Carter-Vac-Filler Equipment for filling of pulverulent products into flexible containers, permeable to gas, as, for example, valve sacks, with the help of a completely closed filling chamber (Carter-Filler)". In comparison, the products of the present invention as defined in the claims herein are hydrophobic, compacted pyrogenic silica where the silica has been compacted by a roller compactor or by a pressing filter belt. The pressing filter belt is described in the US patent 4,877,595 as follows:

The object of the present invention is to provide a method for compressing pyrogenically-prepared, fine-grained silicic acid in a continuous manner. A further object is to assure that the degree of compression of the compressed products which is obtained in the compression device can be retained until the silicic acid leaves the packing device. In accordance with the invention, pyrogenically prepared silicic acid is compressed by means of a rotary vacuum filter which is equipped with a pressing band.

The compacted bulk densities of the silicas of the present invention ranges from 60 g/l to 200 g/l. Compaction obtained by using a roller compactor or a pressing filter belt is not the same as the compaction obtained using the Carter-Filler method.

4. In Table 1 of his present application, it is shown under the headings "AEROSIL R202" and "AEROSIL R805" and "AEROSIL R812" that the silicas have a density of 60/90 which means the silica is available in two different densities; i.e., a density of 60 g/l or 90 g/l. The commercial names of the products would be AEROSIL R202 VV60 and AEROSIL R202 VV90.

5. The AEROSIL silica products obtained in accordance with the invention in this application have a tamped density of approximately 60 g/l which is indicated by the notation "AEROSIL R202 VV60, AEROSIL R805 VV60 and AEROSIL R812 VV60" in Table 1.

6. Similarly, the products identified as AEROSIL R202 VV90, AEROSIL R805 VV90 and AEROSIL R812 VV90 have a density of approximately 90 g/l. as shown in Table 1. The symbol "VV" means the silica has been densified with the pressing filter belt and "V" means the silica has been densified with the roller compactor.

7. The normal products AEROSIL R202, R805 and R812 have a bulk density of 50 g/l. This is shown in Table 1 in the present application. The designation "normal" means a silica that has been compacted by the Carter-Filler method.

8. Thus, the compacted silica products of the present invention which have been compacted using the roller compactor or the pressing filter belt have a greater density than do the silica products known as AEROSIL R202 and AEROSIL 150 shown in our earlier published application '047. In the examples 1-3 of the test results of the '047 patent document, AEROSIL R 202, AEROSIL R 8200 and AEROSIL 150, respectively, are mentioned. For AEROSIL R 202 and AEROSIL 150 the above statement regarding comparative density is correct. However, AEROSIL R 8200 has a tamped density of 140 g/l and is densified using a totally different process (carried out with a ball mill) and has significantly different properties than the mentioned silicas. AEROSIL R 8200 is a reinforcing agent with low thickening properties and cannot be used as a thickener and thixotropic agent for adhesives and sealants in contrast to the other mentioned silicas in the patent application 595,853. In the test results provided in the '047 application, mechanical properties are given but no rheological properties are shown. Furthermore, in the '047 application, the advantage of the shorter incorporation time with

comparable viscosities is not mentioned and demonstrated with examples and data as is shown in the 10/595,853 application.

9. The compacted silicas produced with the roller compactor and filter belt which have a tamped density of approximately 90 g/l show a considerably shorter incorporation time in adhesive and sealant compositions compared to the standard silica products such as AEROSIL R 202 which has a tamped density of approximately 50 g/l. Therefore, the silicas of this invention with a density of 90 g/l can be mixed with and incorporated into adhesive and sealant formulations in a shorter time than is the case with AEROSIL products which have a density of about 50g/l. Also, the silicas with the density of 60g/l exhibit a shorter incorporation time than do silicas with a density of 50g/l.

10. The advantage of the shorter incorporation times and the almost comparable rheological properties could not have been foreseen.

11. As a result, adhesives and sealant compositions can be produced in considerably shorter time using the hydrophobic VV grades of silica and the viscosity of the adhesives and sealants are still within the required and specified viscosity range.

12. When carried out on a commercial scale, the compounding of silica fillers into adhesives and sealant formulations requires time and a shortening of the time required for this operation represents money saved. In addition, there is a special benefit to the use of the method of the present invention which arises from the fact that the results obtained in the laboratory tests which are represented by the data in the present patent application are directly transferable to the commercial scale. Directly transferable means that the technical advantages shown in the laboratory results can be seen also in production trials. As follows there are some tests results of production trials of a thixud polyurethane adhesive with a production dissolver: Incorporation

time AEROSIL R 202 VV 90: 18 minutes and AEROSIL R 202: 25 minutes. Viscosity: adhesive thixed with AEROSIL R 202 VV 90: 73 Pa s, viscosity specification for the adhesive: 80+-10 Pa s.

13. Reference is made to Table 2 in the above-identified application which shows a comparison between the standard AEROSIL R202 silica product and the AEROSIL R202 VV60 and AEROSIL R202 VV90 products which shows that the thickening and thixotropic effect of the AEROSIL R202 VV60 is almost identical to that of the AEROSIL R202 and the incorporation time in seconds is less than the time required for the incorporation of the standard AEROSIL R202 silica into the epoxy resin. Being able to maintain thickening and thixotropic effect while lowering the mixing time is an important technical advantage and was not foreseen in 2003.

An explanation why surprisingly the thickening and thixotropic effect of hydrophobic densified grades can be maintained compared to hydrophilic fumed silica densified grades can be that the interaction forces among the hydrophobic fumed silica particles are mainly Van-der Waals forces. Van-der Waals forces are weaker than hydrogen bridge linkages which are the main interaction forces for hydrophilic fumed silica particles. (By these interaction forces a three-dimensional fumed silica network is created which causes the thixotropy and viscosity increase of the liquid system). As a result, hydrophobic silicas, also higher densified grades, are easier to disperse than hydrophilic fumed silica densified grades and therefore the thickening and thixotropic effect can be almost maintained.

14. As explained in the application, the table in Example 1 shows the incorporation time for all three AEROSIL grades which is a measure of the time in which the AEROSIL is completely wetted with the epoxy resin and has disappeared from the surface of the resin. The

AEROSIL identified as R202 VV90 has the advantage here of a significantly shorter incorporation time as compared to the AEROSIL R202 and AEROSIL R202 VV60.

15. In addition, the dust formation with AEROSIL R202 VV90 is lower than that of AEROSIL R202 and AEROSIL R202 VV60. As an additional advantage I would add that the temperature increase of adhesives and sealants thixed with AEROSIL R 202 VV 90 is lower than with AEROSIL R 202.

16. A copy of my employment history is attached hereto.

I, RÜDIGER NOWAK, hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001, and that such willful false statements may jeopardize the validity of the application, any patent issuing thereon, or any patent to which this declaration is directed.

This 30th day of July, 2009.



Rüdiger Nowak

EMPLOYMENT HISTORY:

Rüdiger Nowak

Education: University degree in chemical-engineering (Fachhochschule Reutlingen, Germany)

1989-1995: Degussa AG, Hanau, Germany: Applied Technology Center : Responsible for fumed and precipitated silicas for polyester resins and adhesives and sealants

1995-2007: Degussa-Hüls AG, Hanau, Germany: Responsible Technical Manager for fumed silica in polyester resins, adhesives and sealants and cable gels for Europe and Asia

2007-2009: Evonik Degussa GmbH, Rheinfelden, Germany: Technical Service Manager for fumed and precipitated silicas, carbon black and silanes in adhesives and sealants in Europe